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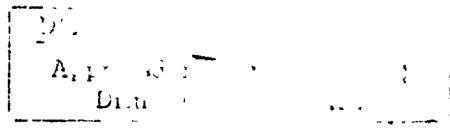
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Technical Report No. 2

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FRACTO-EMISSION ACCOMPANYING ADHESIVE FAILURE

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10 by  
 J. T. / Dickinson, M. K. / Park / E. E. / Donaldson / L. C. / Jensen

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)	Experimental studies of the emission of electrons (EE), positive ions (PIE) and photons (phE) accompanying fracture are presented for systems involving adhesive failure. The systems studied include interfacial failure between epoxy--brittle materials, polybutadiene-glass, and pressure sensitive adhesives--polymers. Time distributions and energy distributions are shown and for a few systems we compare EE and phE.	

## FRACTO-EMISSION ACCOMPANYING ADHESIVE FAILURE

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### ABSTRACT

For a wide range of materials, emission of electrons (EE), positive ions (PIE), and photons (phE) is observed accompanying fracture. Here we report on work concerning emission from adhesive failure. The systems studied include interfacial failure between epoxy--brittle materials, polybutadiene-glass, and pressure sensitive adhesives--polymers. When interfacial failure occurs, it produces highly excited surfaces with intense charge separation. De-excitation in the presence of surface charge produces intense, long lasting (several minutes) emission. Energy distribution curves for both EE and PIE decrease monotonically with a tail extending out to several hundred eV. For a few systems we compare EE and phE which show different decay characteristics.

## INTRODUCTION

During and following the fracture of materials, the emission of charged particles,<sup>1-6</sup> neutral particles,<sup>7</sup> and photons<sup>3,8,9</sup> have been observed. Studies of electron emission (EE) during the tensile elongation of oxide-coated aluminum, sometimes referred to as tribo-stimulated exoemission, have shown that fracture of the oxide coating is the initial cause of the ejected electrons.<sup>4-6</sup> Similarly, positive ion emission (PIE) neutral emission<sup>6</sup> and photon emission<sup>3</sup> have been observed on the same oxide-aluminum systems, and were shown to be due to oxide fracture. (Photon emission is most frequently referred to as triboluminescence.<sup>8,9</sup>) These various types of emission have some shared characteristics suggesting common mechanisms in their production. We refer to all forms of such emission accompanying fracture as "fracto-emission" (FE).

In Ref. 10 we reviewed the factors which appear to contribute to FE and presented a survey of charged particle measurements we made on a wide range of materials undergoing fracture. Briefly, when a crack propagates through a material the crack walls are left in a highly excited, nonequilibrium state. For nonmetals this departure from equilibrium involves broken bonds, liberated fragments (e.g. free radicals, atoms, charged species) defects (e.g. point defects), charge separation often involving the filling of localized surface states and intense electric fields) and a localized rise in temperature. All of these factors represent high concentrations of energy in a small volume and may contribute to fundamental processes such as Auger transitions,<sup>11</sup> ESD-like transitions,<sup>12</sup> and dipole-transitions that lead to FE. An example of this is chemi-emission<sup>13</sup> where during a chemical reaction at a surface an excited intermediate state is produced capable of producing either photons or electrons, the latter by an Auger process (and in our case would involve trapped electrons closest to the vacuum level).

During fracture at interfaces, often called adhesive failure or interfacial failure, the bonds broken and degree of charge separation may be quite

different from cohesive failure in a homogeneous material. For example, Jonath<sup>14</sup> discusses the fact that as one approaches the adherend, there is a gradient in chemical and mechanical structure due to an influence of the bonding surface. Thus, upon failure, depending upon the locus of fracture, unique excitations and species can be produced. Also, in polymers, it is known that fracture can produce high concentrations of free radicals, as determined by e.s.r. measurements.<sup>15</sup>

In this paper we present additional work on FE accompanying and following the fracture of materials where interfacial failure occurs for systems involving a polymeric component (e.g. matrix, adhesive, or adherend).

#### EXPERIMENTAL

Details of our experimental procedures are given in Ref. 10. The samples are fractured by applying tensile stress. The typical cross-sections of bulk samples were 0.1 to 20 mm<sup>2</sup>. We fractured bare filaments, typically 10  $\mu\text{m}$  in diameter, of such materials as glass and Kevlar. These were mounted so that single filaments could be broken sequentially. Epoxy-strands of these materials consisted of about 250 filaments in DOW DER 332 Epoxy.

Filled elastomers consisted of untreated small glass beads 30-95  $\mu\text{m}$  in diameter in a polybutadiene matrix. These samples had cross-sections of 15 mm<sup>2</sup> and contained amounts of glass beads varying from 0 to 34% by volume.

Another type of interfacial failure studied involved the delamination of 3M Filament Tape; i.e., the separation of the polyester backing from the glass filaments which adhere by means of a natural rubber-based adhesive. For such studies, typically 1 cm<sup>2</sup> of surface was created in a time of 10 s. Similar studies were made from the peeling of 3M MAGIC TRANSPARENT TAPE from a PMMA surface.

The charged particle experiments were carried out at  $1-4 \times 10^{-6}$  Torr and the residual gases consisted primarily of CO, H<sub>2</sub>, and CO<sub>2</sub>. Some samples were tested in an ion-pumped system at  $10^{-8}$  Torr--no differences in the FE were

observed in the two environments. Charged particles were detected with channel electron multipliers (CEM), Galileo Electro-Optics (4039), positioned 1 cm from the sample. Photons were detected using a Bendix BX 754A Photon Countertube with an S-20 photo-sensitive surface. Data were handled with standard pulse counting methods and stored in a multichannel analyzer.

## RESULTS

The fracture of filaments of graphite and Kevlar gave rapidly decaying EE with time constants on the order of 10  $\mu$ s for graphite and 100  $\mu$ s for Kevlar. The graphite decay is very similar to that observed for E-Glass and S-Glass filaments.<sup>10</sup> We also fractured pure DER 332 epoxy and obtained the 25  $\mu$ s decay time as shown in Fig. 1. PIE time distributions for these materials are indistinguishable from these electron curves.

When we fracture the corresponding fiber-reinforced epoxy strands made from the same filaments and epoxy, we find emission curves that differ from those for pure materials. Figure 2 shows EE and PIE curves for the Kevlar-Epoxy and Graphite-Epoxy systems. On the scale shown, the time required for fracture is usually less than one channel. The decay is seen to last for many seconds--some of the more intense emitters will give detectable emission an hour after fracture. Although intensities differ, we have simultaneously measured PIE and EE from the same fracture event and found the decay curves to be of the same shape. This suggests a common rate-limiting step for the two types of emission. SEM photographs of the samples show delamination and separation of the filaments from the epoxy. This interfacial or adhesive failure is most likely responsible for these major FE components with the slow decay and may serve as an indicator of the extent of interfacial failure that has occurred. Figure 3 shows on a log scale the normalized energy distribution of the EE and PIE produced by differentiation of retarding potential curves. Both curves are very similar showing a peak near 0 eV with a significant quantity of higher

energy particles in the tail. The presence of these higher energy particles suggests that the charging of the fracture surface (due to separation of charges) plays an important role in producing the observed charged FE. Again, the similarities in the EE and PIE energy distributions provides further support that they share a common mechanistic step.

Similar results are seen for polybutadiene with and without glass beads as seen in Fig. 4. The peak intensity created during fracture and the after-emission are considerably more intense when the glass beads are present. Gent<sup>16</sup> has shown that the beads become detached during straining of the matrix. This adhesive failure is considered the cause of the enhanced FE. Figure 5 shows the dependence of the total emission (in 200 s) as a function of the quantity of glass beads in the polybutadiene, for both EE and PIE increasing with increasing bead concentration.

The peeling of tape from a surface also produces charged particle emission as well as visible photons. Figure 6 shows the EE and phE from peeling 3M MAGIC TRANSPARENT TAPE from Plexiglas. The EE is high during peeling and decays slowly afterwards. The phE is above the noise only during peelings. Although these FE components may share a common excitation step, the photon mechanism decays away much quicker. Similar results are seen for the delamination of the filaments from the polyester backing of 3M FILAMENT TAPE, as seen in Fig. 7. Between a and b we have delaminated the surfaces, and reattached the surfaces at c (which causes the EE to disappear), then repeated the process, showing that EE and phE is produced by the second and third detachment (i.e., virgin bonds are not necessary).

#### CONCLUSIONS

We have shown that fracture involving adhesive failure leads to intense, long-lasting EE and PIE. This has been shown for fiber-reinforced epoxy, polybutadiene filled with glass beads, and pressure sensitive adhesives. The

detachment of adhesives from adherends is frequently accompanied by intense charge separation<sup>14</sup> as well as the creation of excited and reactive species, e.g., free radicals. As previously argued,<sup>10</sup> we do not attribute the observed charged particle emission to field emission, but we do feel that the electric field from surface charge patches (of both signs) is responsible for the particle kinetic energies observed and may also affect the emission intensity. The latter could be due to an influence on the mobility of active species near or at the fracture surface as well as a possible E-field dependence of transition probabilities in excited species. The enhanced effects we see when interfaces are involved is likely due to a) the different types of chemical species produced by adhesive failure (as compared to cohesive failure) and b) the degree of charging that occurs with interfacial failure.

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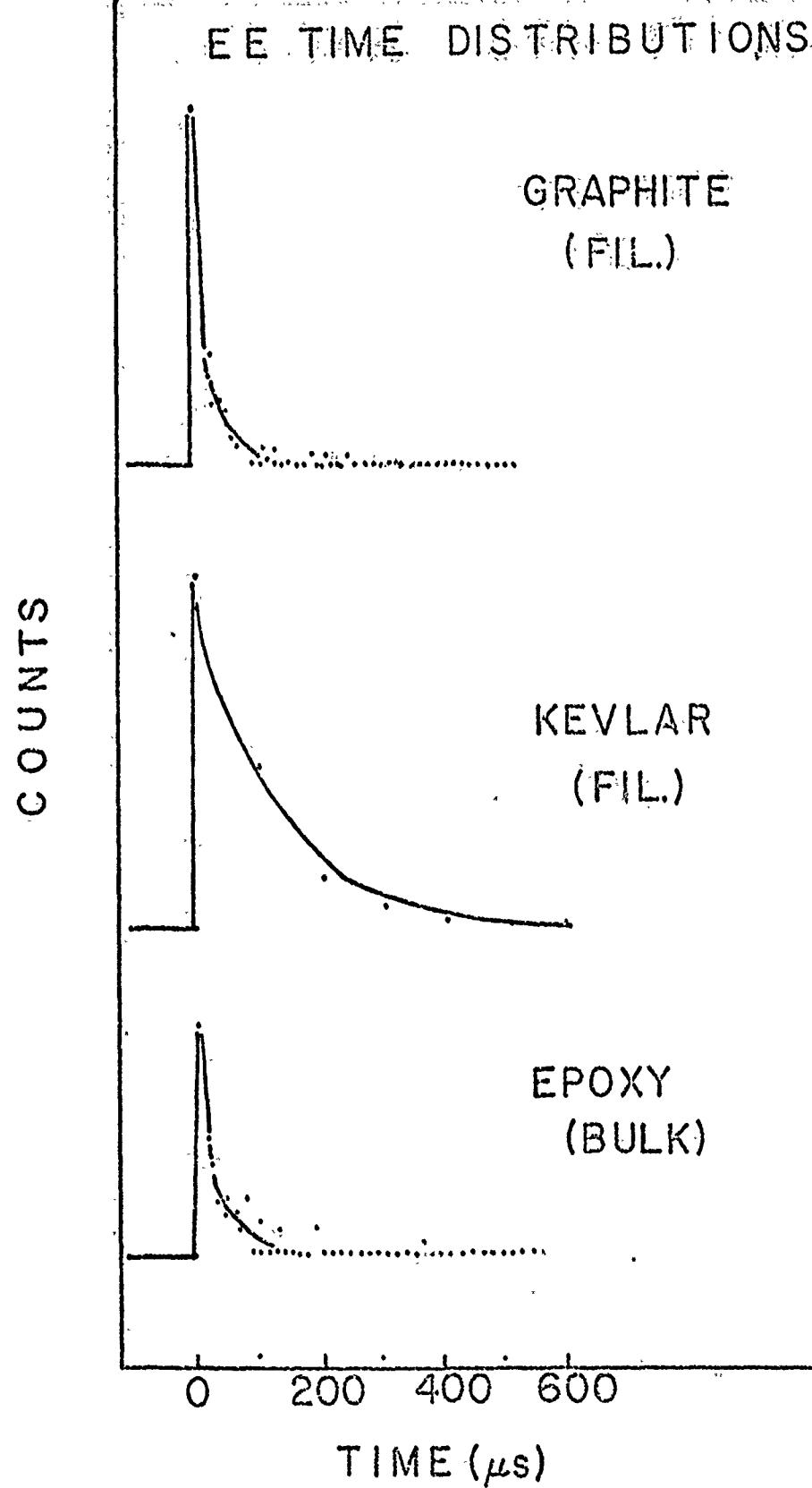
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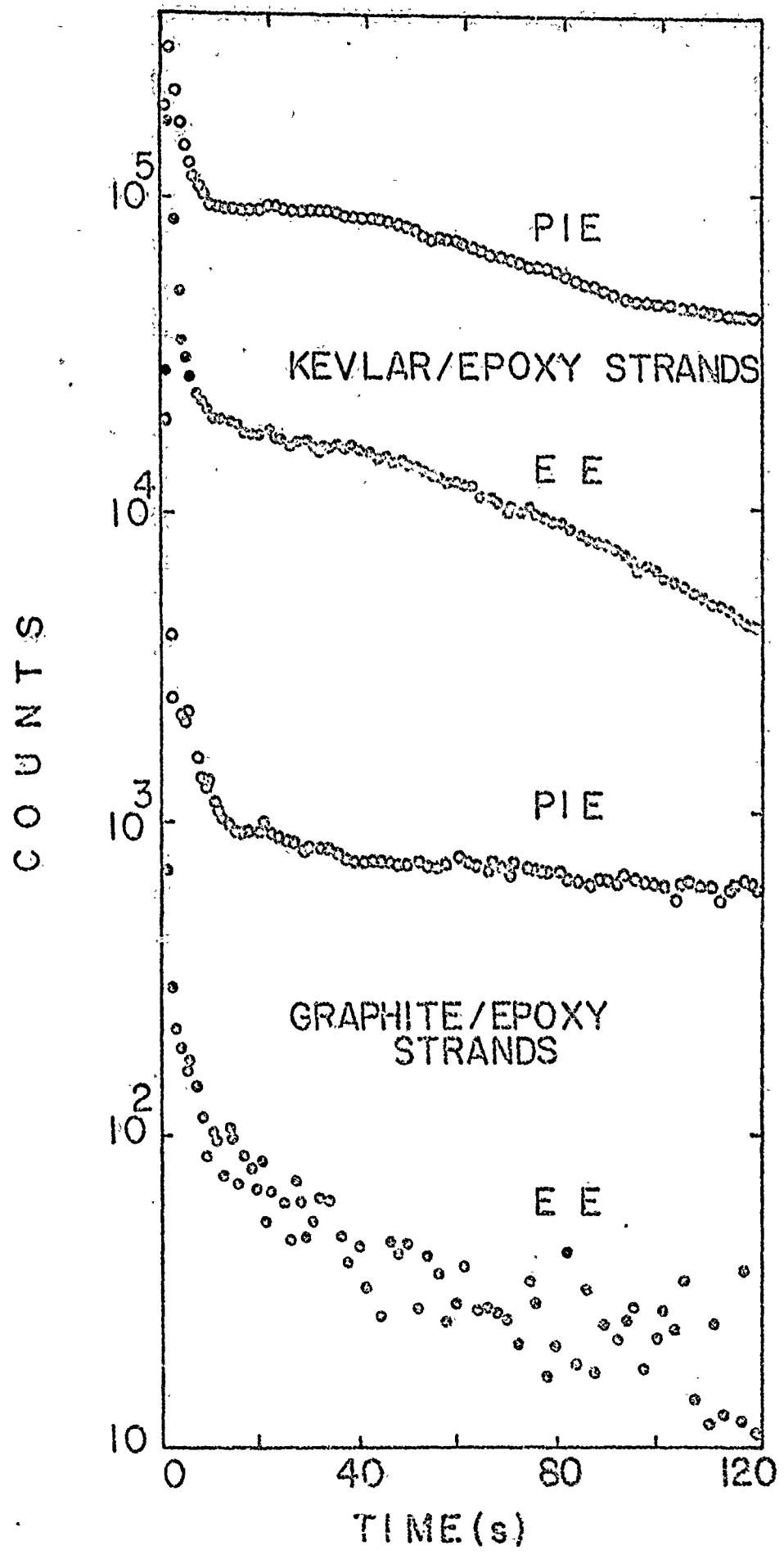
### Figure Captions

- Fig. 1. Time distributions of EE due to fracture of 10  $\mu\text{m}$  graphite and Kevlar filaments and bulk epoxy (Dow DER 332).
- Fig. 2. EE and PIE from fracture of filament/epoxy strands; Kevlar and graphite.
- Fig. 3. Energy distributions, on a log scale, for EE and PIE from Kevlar/Epoxy.
- Fig. 4. EE and PIE from fracture of polybutadiene with and without glass beads.
- Fig. 5. The total emission (counts accumulated over 200 s) as a function of the volume percent of glass beads in polybutadiene.
- Fig. 6. EE and Photons from peeling 3M-MAGIC TRANSPARENT TAPE from PMMA.
- Fig. 7. EE and Photons from Delamination of 3M FILAMENT TAPE.

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EE AND PIE ENERGY DISTRIBUTIONS  
FROM KEVLAR-EPOXY STRANDS

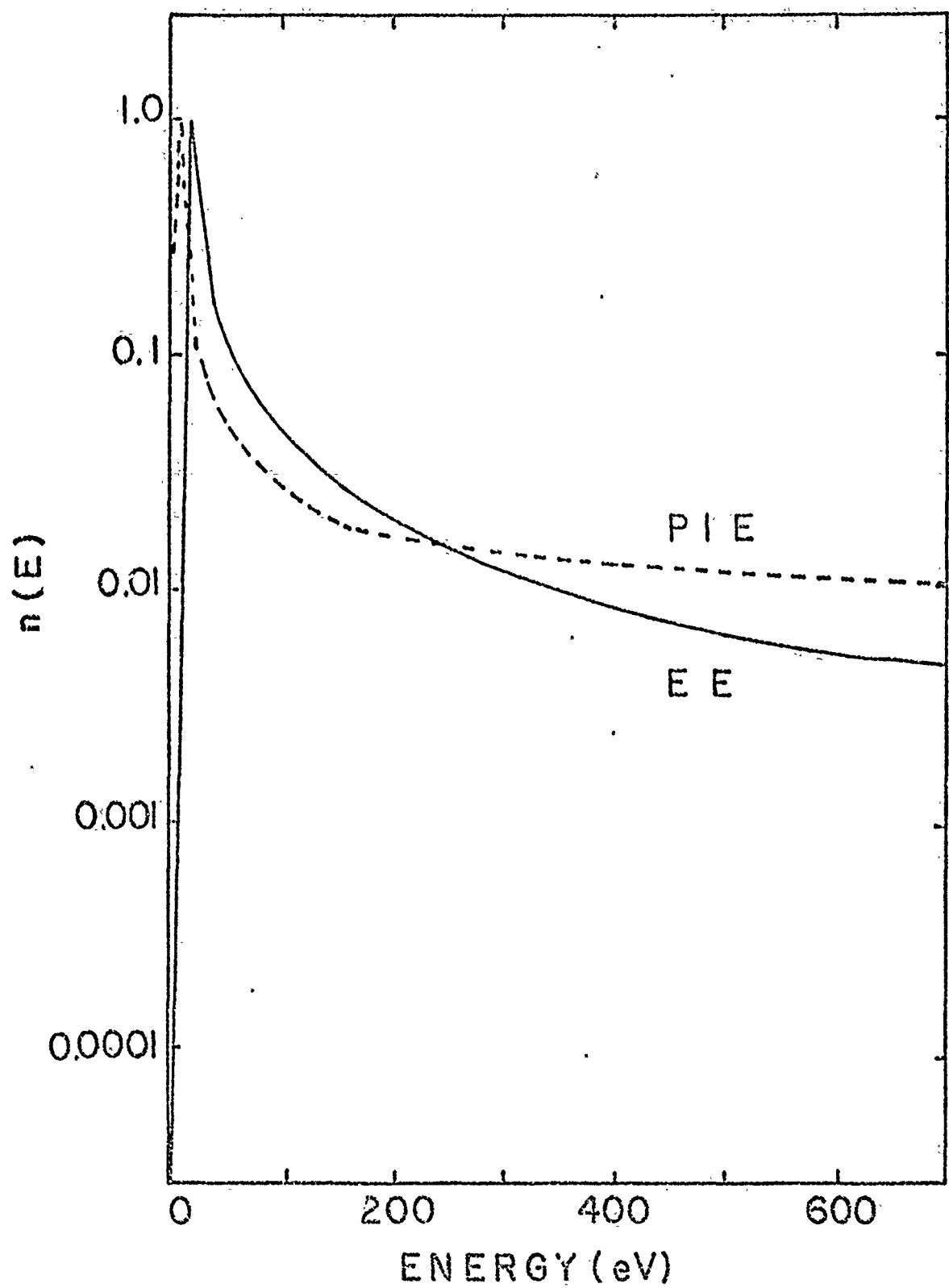


Fig. 3

## FE FROM POLYBUTADIENE

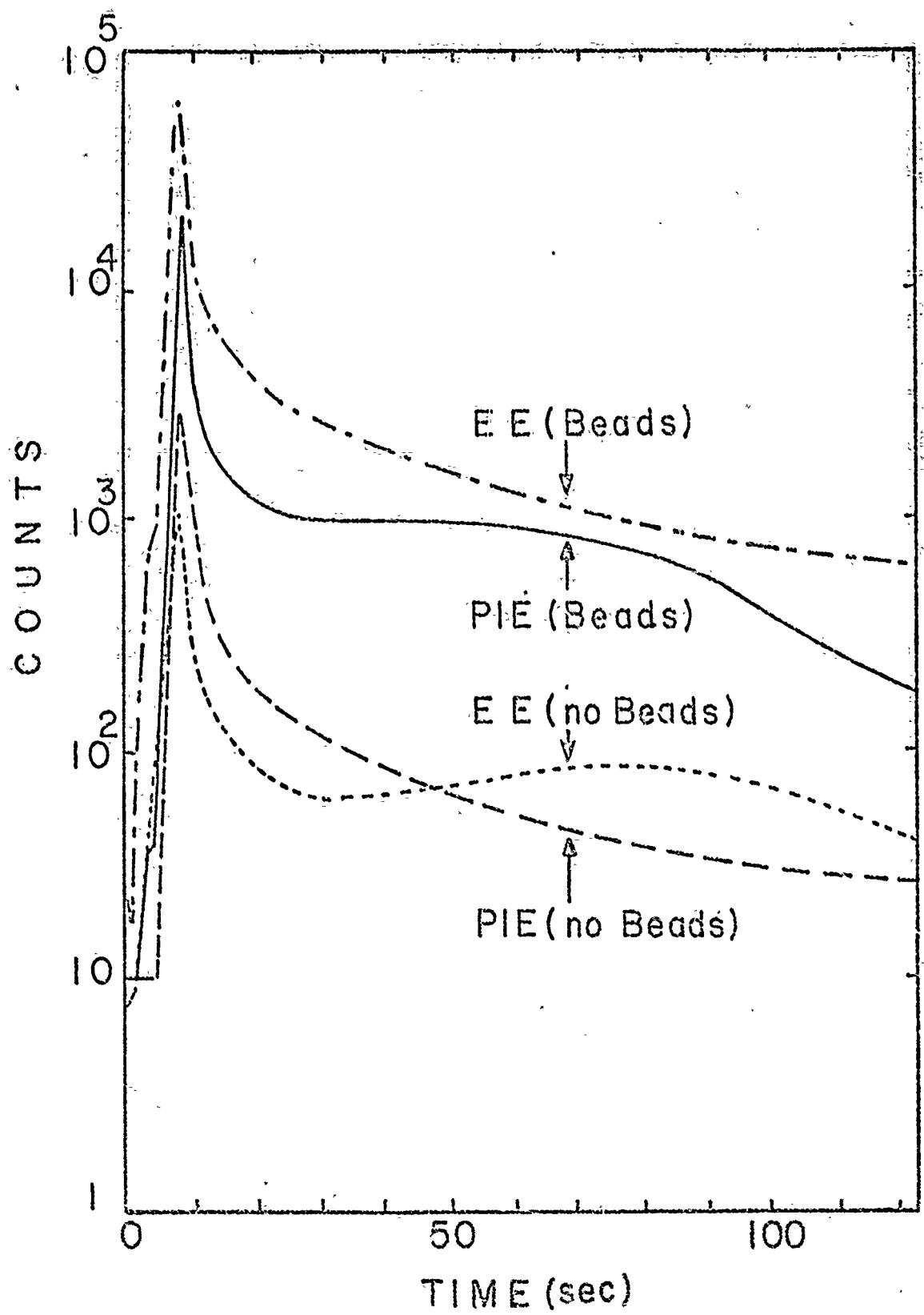


Fig 4

POLYBUTADIENE CONTAINING  
GLASS BEADS 30-95  $\mu\text{m}$  DIAM.

Particle Emission, units  $10^6/\text{cm}^2$

10  
5  
0

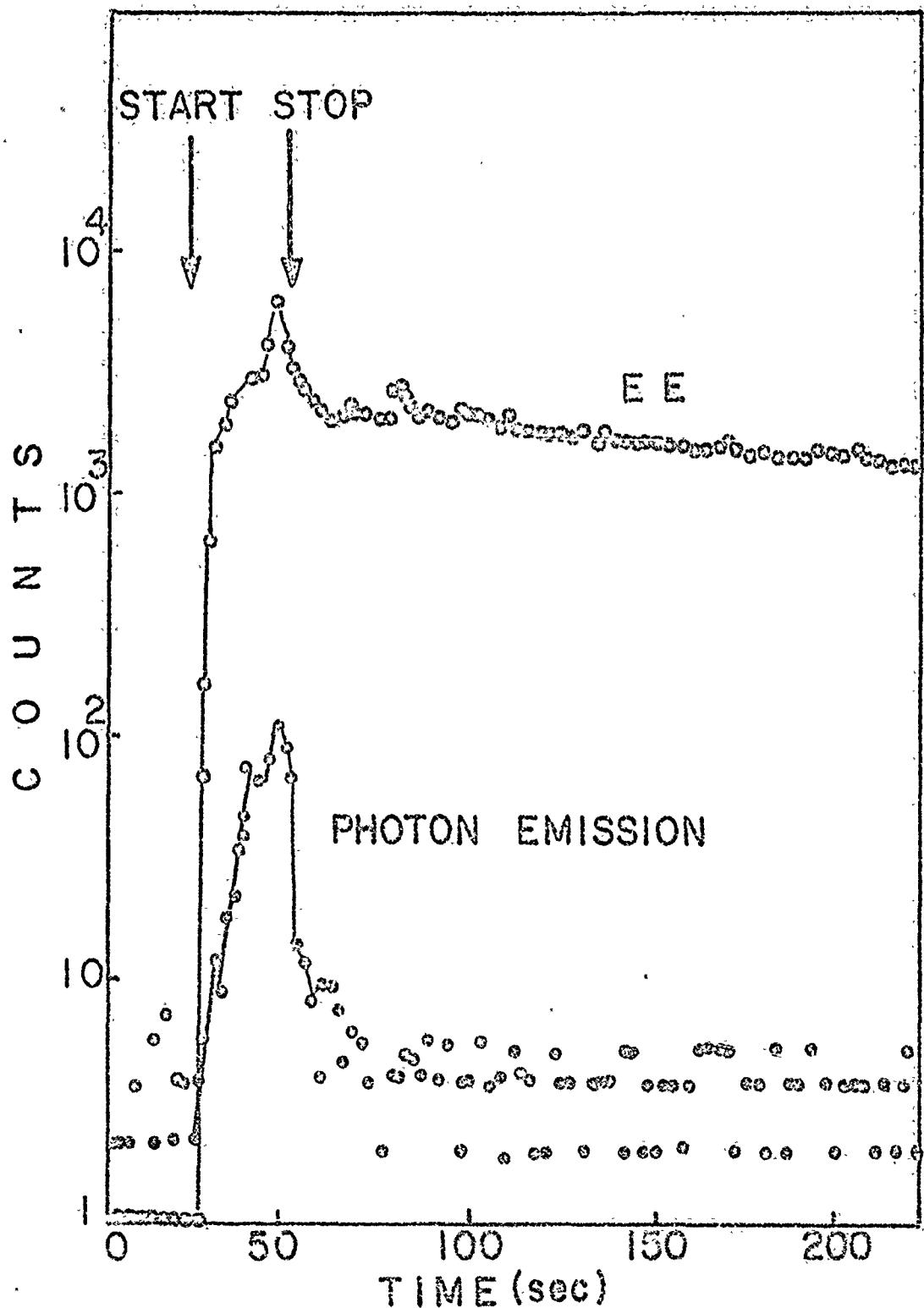
Total Positive  
Ion Emission  $\oplus$

Total Electron Emissions  $\ominus$

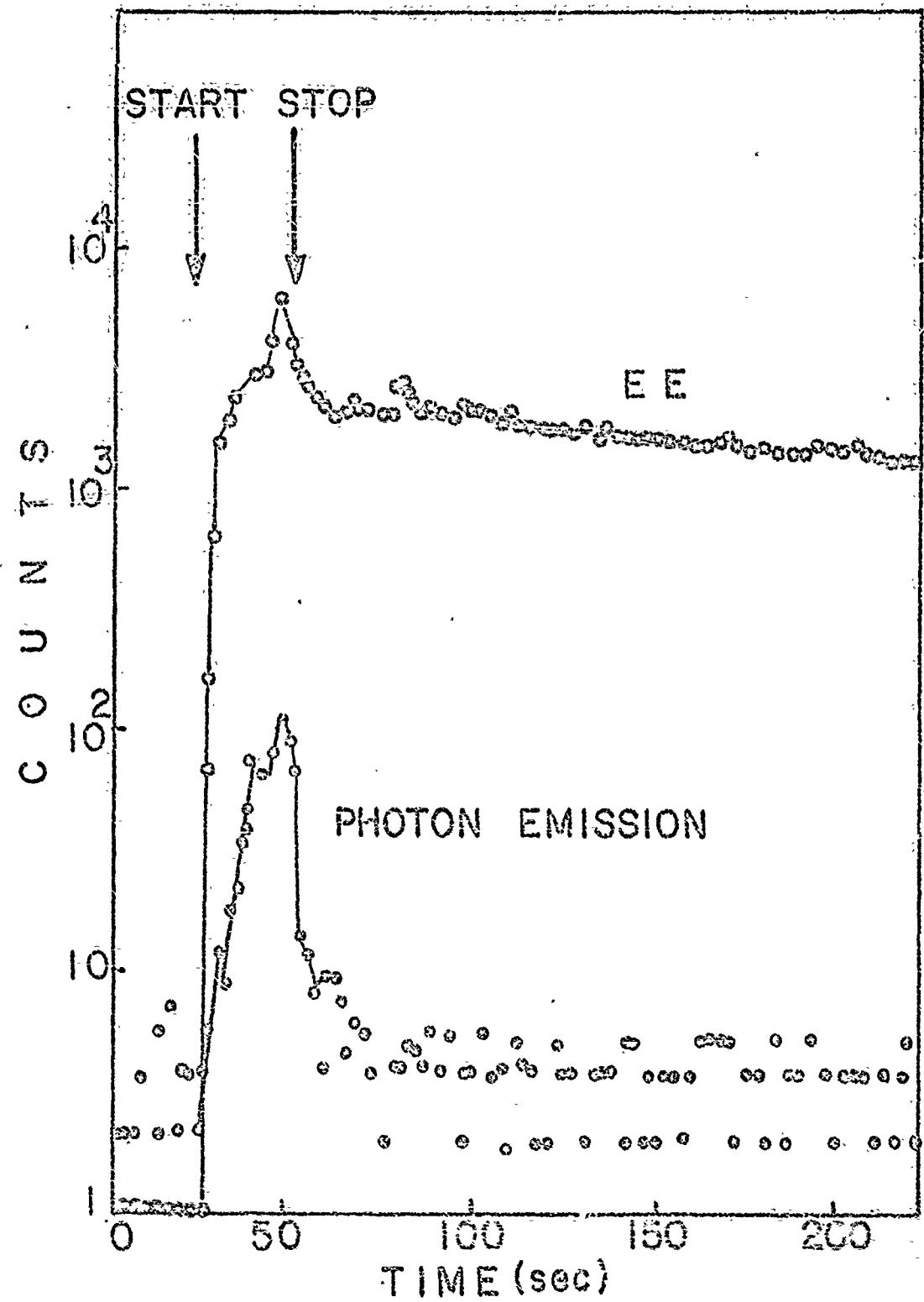
30  
20  
10  
0

Volume % Glass Beads

EE AND PHOTON EMISSION FROM  
PEELING 3M-MAGIC TAPE FROM PMMA



EE AND PHOTON EMISSION FROM  
PEELING 3M-MAGIC TAPE FROM PMMA



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